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Edelson, Burton I.; Robertson, W.D.

Yale University

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Burton I. Edelson
and
W. D. Robertson

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THE EFFECT OF A SURFACE OXIDE FILM

ON TORSIONAL RELAXATION

BURTON I. EDELSON
and
W. D. ROBERTSON

29 September 1954

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Via: Commanding Officer, Naval Damage Control Training Center, U. S. Naval Base, Philadelphia 12, Pennsylvania

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THE EFFECT OF A SURFACE OXIDE FILM ON TORSIONAL RELAXATION*

B. I. EDELSON† and W. D. ROBERTSON‡

The effect of a surface oxide film on the torsional relaxation of polycrystalline cadmium wires was investigated. The phenomenon of reversal when the film is removed during relaxation, known as the abnormal after-effect, is shown to be a reproducible effect directly dependent on the presence and elastic properties of a surface film. Various physical parameters, such as metallurgical history of the metal, film conditions and time functions, are varied in relaxation tests to show this dependence. An explanation of the abnormal after-effect is given in terms of a two-component mechanical model. An equivalent explanation of the abnormal after-effect may be given in terms of the dislocation barrier theory.

L'EFFET D'UNE COUCHE D'OXYDE À LA SURFACE, SUR LA RELAXATION EN TORSION

On a investigué l'effet d'une couche d'oxyde à la surface, sur la relaxation en torsion de fils polycristallins de cadmium. Il est montré que le phénomène d'inversion, quand la couche est enlevée pendant la relaxation, connu comme la répercussion anormale, est un effet reproductible qui dépend directement de la présence et des propriétés élastiques de la couche à la surface. Divers paramètres physiques, tels que l'histoire métallurgique du métal, les conditions de la couche et des fonctions du temps sont variés dans les essais de relaxation, en vue de montrer cette dépendance. Une explication de la répercussion anormale est donnée en termes d'un modèle mécanique à deux composantes. Une explication équivalente peut être donnée en termes de la théorie de barrière de dislocations.

DER EFFEKT EINES OBERFLÄCHENOXYDFILMES AUF DIE TORSIONSNACHWIRKUNG

Es wurde der Effekt eines Oberflächenoxydfilmes auf die Torsionsnachwirkung von polykristallinen Kadmiumdrähten untersucht. Es wird gezeigt, dass die als "anomale Nachwirkung" bekannte Erscheinung der Umkehrung beim Ablösen des Filmes während der Relaxion ein reproduzierbarer Effekt ist, der direkt von der Anwesenheit und von den elastischen Eigenschaften des Oberflächenfilmes abhängt. Um dieses Abhängigkeitsverhältnis zu zeigen wurden verschiedene physikalische Parameter, wie die metallurgische Vorgeschichte des Metalls, die Filmeigenschaften und die Zeitkonstanten in den Nachwirkungsversuchen variiert. Es wird eine Erklärung der anomalen Nachwirkung im Rahmen eines mechanischen Zweikomponentenmodells gegeben; eine entsprechende Erklärung der anomalen Nachwirkung kann auf Grund der Theorie der Versetzungshindernisse gegeben werden.

Introduction

If a metallic wire is twisted beyond its elastic limit and the applied stress is released, there occurs an instantaneous elastic recovery followed by a time-dependent relaxation. This relaxation, called the normal after-effect, is such that the wire recovers, approximately, an equal number of angular units in equal increments of the logarithm of time [1]. When recovery in degrees is plotted against log time a straight line is obtained, except near zero time and near infinite time.

It was suggested by Cottrell [2] that, in accordance with the current concept of plastic deformation, an elastic film on the surface of a wire constitutes a barrier to those dislocations associated with torsional deformation that ordinarily pass through the external surface and, accordingly, they should concentrate beneath the surface film. The extent to which the dislocations are blocked defines the residual elastic stress associated with the torsional deformation. After removal of the applied stress the wire untwists in accordance with the normal after-effect. If, during the untwisting, the surface film is quickly removed—by etching, for example—the dislocations still concentrated at the surface are released and they escape from the metal; this corresponds to a motion in the direction of the original deformation, opposite to the existing motion, and should manifest itself by a change in the rate of the normal after-effect. Barrett [3; 4] observed this phenomenon in single crystals of zinc and in polycrystals of iron, zinc and cadmium. In some cases the wire actually reversed (moved in direction of original twist) after removal of the surface layer. This reversal Barrett termed an "abnormal after-effect." In other cases the untwisting was merely slowed and the resultant plot showed a decrease of slope.

In the first paper [3] Barrett established the existence of the effect in zinc and iron. Subsequent work [4] indicated: (1) the abnormal effect did not originate from a thermal transient or acid attack on the metal itself; (2) the sensitivity of the method

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†Lieutenant, U.S.N., Hammond Metallurgical Laboratory, Yale University, under sponsorship of the U.S. Naval Postgraduate School, Monterey, California.

‡Hammond Metallurgical Laboratory, Yale University.

was great enough to detect very thin films (those formed in air at room temperature in less than one hour); (3) that moderate plastic deformation did not disrupt the coherency of the film to the extent of eliminating the effect; and (4) that prior cold work increased the magnitude of the normal after-effect.

In addition to the preceding interpretation of the abnormal after-effect, there is an alternative, or equivalent, explanation that is generally consistent with the experimental facts reported by Barrett, namely, that a coherent elastic film on the surface of a plastically deformed wire constitutes a two-component mechanical system. Twisting the wire and film produces an elastic stress at the surface causing the wire to recover at a greater rate than it would in the absence of a film. The sudden removal of the film allows the wire to seek its own recovery rate, even reversing temporarily to the position it would have assumed in the absence of the film. The investigation described below was designed to evaluate this hypothesis in terms of new experiments and to determine how far the two interpretations can be considered equivalent.

The following determinations are necessary to obtain the data required to form a self-consistent set from which unambiguous conclusions can be formulated:

(1) The difference in relaxation behaviour of an oxidized wire and a clean wire, including the effect of etching.

(2) The effect of previous history of the metal (cold-working and annealing) on the abnormal after-effect.

(3) The effect of thickness of the oxide film.

(4) The effect of holding time prior to release of load.

(5) The effect of a delay in removing the surface film.

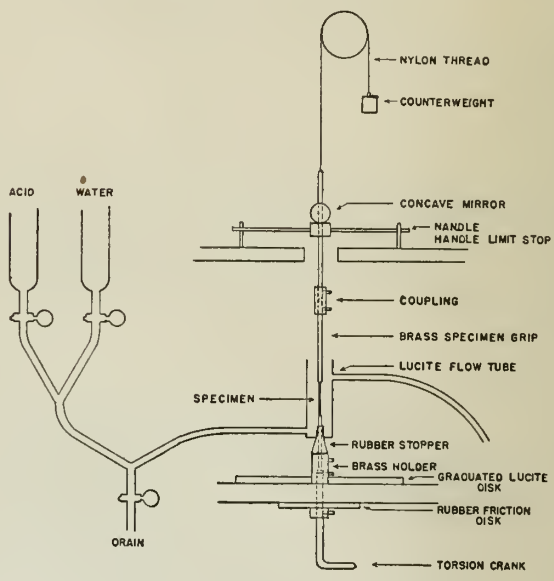
(6) The effect of twisting a clean wire and then applying the film after twisting.

Experimental Procedure

For preliminary work the apparatus developed by Barrett [4] was used. Several metals were tested with various cold-working and annealing treatments, types of surface films, and concentrations of acids to obtain normal and abnormal after-effects as distinct as possible. This preliminary work showed that polycrystalline cadmium with an anodic film, and dilute sulfuric acid as etchant, was the most effective of the combinations tested.

For quantitative work it was necessary to employ precision timing; to eliminate accidentally introduced bending, tensile and compressive stresses; to control the exact amount of twist and the time of holding after twist; to eliminate possible buoyancy, wetting and thermal transient effects; and finally, to provide for the application of a surface film subsequent to twisting.

The apparatus shown in Figure 1 was developed



SCHEMATIC DRAWING OF APPARATUS

FIGURE 1. Schematic drawing of apparatus.

to meet the above requirements. The wire specimens, each 5.6 cm long and 1.11 mm in diameter, were cemented in brass grips with a DeKhotinsky-type wax having a softening point of about 140°C. Length of specimens between grips was 4.0 cm. No evidence was found of plasticity in the cement. The bottom grip was rigidly fastened to a graduated lucite disk and to the crank used for twisting the wire. Amount of twist was determined by matching the graduated disk with a line on the base of the apparatus. The top grip was rigidly coupled to a handle and concave mirror. The assembly was maintained vertical by a nylon thread and counterweight, resulting in a net tensile force of 1.3 gm. The specimen was enclosed by a lucite tube through which water or the acid etchant could be circulated. The bottom grip passed through a rubber stopper which remained stationary during the twisting operation. Two burettes were connected to the inlet of the flow tube and they permitted changing of the medium from water to acid, as desired. A drain valve and overflow tube were also provided; in this way liquid level remained constant and the

specimen was always immersed which precluded significant temperature changes or other effects due to immersion.

The apparatus and following procedure permitted the wire to be twisted, held, released and its relaxation observed with a minimum of handling. When the crank was turned, the grip holder, lucite disk, specimen and its grips, and the handle and mirror turned with it; the flow tube and rubber stopper remained stationary. If the crank were turned far enough, the handle encountered two handle limit stops fixed to the top of the apparatus which prevented its further turning. This fixed the top of the specimen, upper specimen grip and mirror in the "zero position". Further turning of the crank twisted the specimen. When the specimen was twisted the required amount and held for the prescribed time, the crank was reversed until the handle was free of the limit stops; zero time was marked; and the specimen was allowed to relax. Since a rubber friction disk held the crank, holder and bottom grip stationary, untwisting of the wire was transmitted to the mirror via the upper grip. A galvanometer lamp and scale together with an electric timer, were used to record the movement of the mirror.

Commercially pure cadmium, obtained from the New Jersey Zinc Company, was used. Specimens were prepared by casting into bars, rolling and then drawing to the same final diameter (1.11 mm). Unless otherwise indicated, specimens having a grain size about 1/100 of the wire diameter were used in the cold-worked condition, produced by approximately 60 per cent reduction in area. Experiments with wires at various times after drawing demonstrated that the effect of annealing at room temperature was negligible.

Surface films were produced in a cell consisting of the specimen as anode, a platinum cathode, and a 1N sodium hydroxide electrolyte. It was found that 2.5 volts (0.02 amp/cm^2) gave a very satisfactory anodic film in 20 minutes. The film formed was cadmium hydroxide [5], which is insoluble in water but dissolves readily in 2 per cent sulfuric acid.

The standard procedure was to prepare wires of the final diameter about 6 feet long and then wash with organic solvent, acid, water and alcohol to remove traces of lubricant and foreign matter. The wires were then cut to the standard length (5.6 cm), anodized, cemented in grips and then tested in the apparatus. During the tests the liquid media, always at room temperature, were allowed to circulate slowly through the flow tube to maintain constant conditions and dissipate heat. All runs were made at room temperature (23°C to 25°C). All wires were twisted 180 degrees in approximately 4 seconds. The etchant used in every case was 2 per cent H_2SO_4. The ends of the grips were coated with paraffin to prevent galvanic action between the acid and the brass grips. Unless specifically stated otherwise the following parameters were kept constant. The time from commencement of twist to release of load was 30 seconds. The wire was twisted, held, and allowed to relax while surrounded by distilled water. The etchant was applied at 400 seconds after release of load.

Experimental Results

Effect of Anodic Film

An investigation was made to determine the effect of an anodic film on the relaxation behaviour of cold-worked polycrystalline cadmium. The tests were conducted in quadruplicate in order to determine the degree of reproducibility. Four specimens were anodized in the standard manner, and four were etched in 2 per cent H_2SO_4 for five minutes prior to the run. The resulting data are given in Figure 2 where strain (in degrees) is plotted as a function of log time. The figures in parenthesis represent the normal after-effect strain rate, ex-

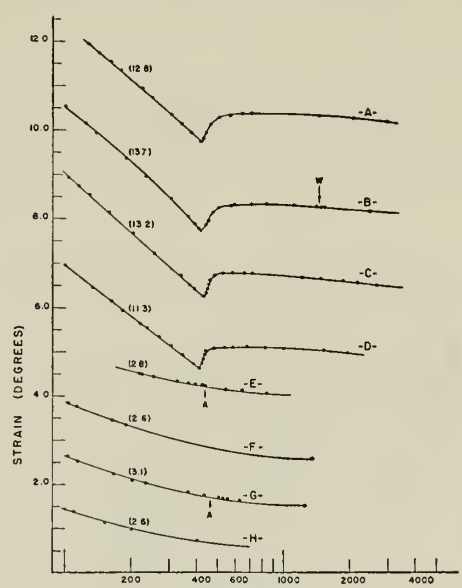


FIGURE 2. Comparison of after-effect curves of anodized and nonanodized specimens. Curves are in quadruplicate: –A– through –D– were anodized; –E– through –H– were not anodized. *W* indicates water added. *A* indicates acid added. Figures in parenthesis indicate strain rate (in tenths of a degree recovered between 200 and 400 seconds).

pressed in tenths of a degree strain recovered between 200 and 400 seconds.

The abnormal after-effect is very clearly demonstrated and associated with the removal of the film. Prior to the removal of the film, the slope of oxidized specimens is linear and the deviation of the slope is less than 10 per cent from the mean. The magnitudes of reversal in tenths of a degree are 6.4, 5.5, 5.4 and 4.9, respectively. The behaviour of the etched wires is markedly different; the strain rate is only about one quarter of that obtained with anodized wires and is not linear. Although acid replaced the control media, water, at points marked *A* on curves -E- and -G-, no effect was observed. Water replaced acid at point marked *W* on curve -B-. Again no effect was noticed. At no time, in this or succeeding experiments, was acid attack of the metal indicated, either by formation of bubbles or by change in diameter of the specimen after the run.

Effect of Cold Working

Polycrystalline cadmium wires (all 1.11 mm in diameter) were identically prepared except for the amount of cold work following a full anneal of one hour at 250°C. After standard anodizing treatment these wires were tested to determine the effect of cold-working on the normal and abnormal after-effects and the results are shown in Figure 3. The specimen representing zero per cent reduction in area was annealed for 1 hour at 250°C after the final drawing operation.

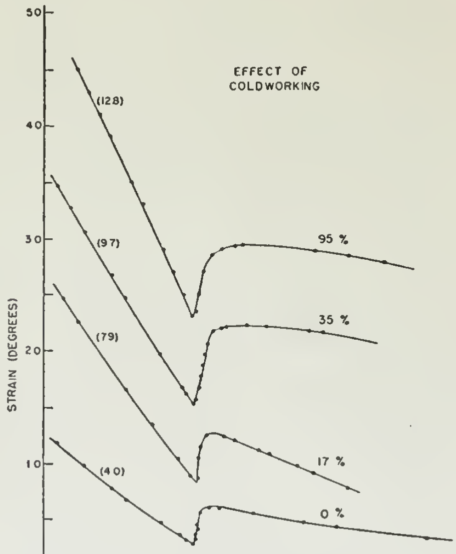


FIGURE 3. Effect of cold work on after-effect curves Percentages indicate amount of reduction in area after last anneal. 0 per cent cold work indicates 250°C anneal after drawing.

As might be anticipated, increasing amounts of cold work increase the normal strain rate when the anodic coating is the same. Likewise, increasing the amount of cold work has a tendency to increase the magnitude of reversal, in fact it is 3.3, 4.5, 6.9 and 6.4 tenths of a degree from zero to 95 per cent reduction, respectively.

Effect of Annealing Temperature

Using the specimens in the highly cold-worked condition (95 per cent reduction in area) a series of runs were made to determine the effect of subsequent annealing. Accordingly, specimens of this wire were annealed in air at various temperatures for one hour, then anodized in the standard manner. The plots of strain *versus* log time are given in Figure 4. Here the initial strain rate became less

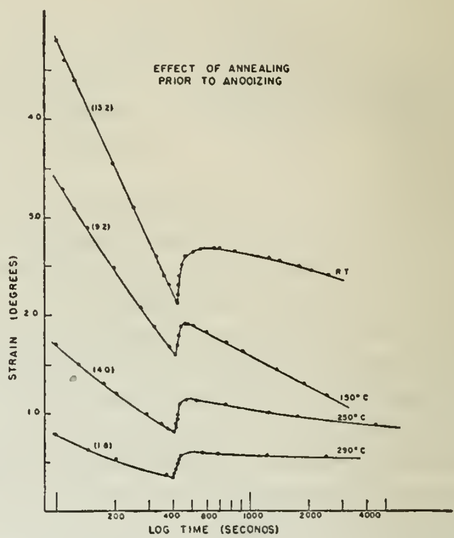


FIGURE 4. Effect of annealing for one hour at various temperatures on after-effect curves.

with increasing annealing temperature. The amount of reversal (in tenths of a degree) also decreased with increasing annealing temperature. These two quantities are plotted in Figure 5.

The effect of annealing, as well as that of cold-working, demonstrates the influence of the metallurgical history of the wire on its relaxation behavior. These results might have been predicted on the basis of mechanical properties, by noting that increasing annealing and decreasing cold work both facilitate plastic deformation, leaving a smaller portion of the original twist recoverable and therefore decreasing the amount and rate of relaxation.

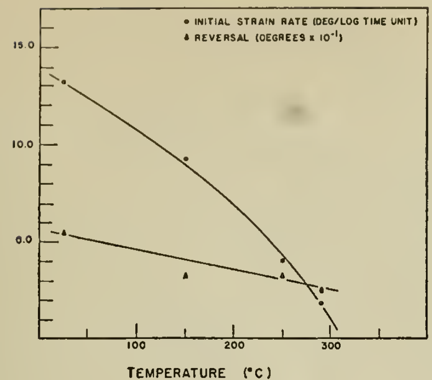


FIGURE 5. Effect of annealing at various temperatures for one hour prior to anodization on initial strain rate and amount of reversal.

Effect of Thickness of Oxide Film

A series of runs was made to determine the effect of thickness of the anodic film. Successive specimens of cold-worked wire were anodized for various lengths of time, all at 2.5 volts. The results are shown in Figure 6. Differences in anodizing time could be noted visually: the one-minute film was a blackish-brown (probably mostly oxide); films produced by longer anodizing times were successively lighter (hydroxide); and the 40-minute film was a very light gray, appearing slightly spongy. The nonanodized specimens again showed no reversal upon application of acid (applied at *A* in Figure 6). The one-minute specimen showed

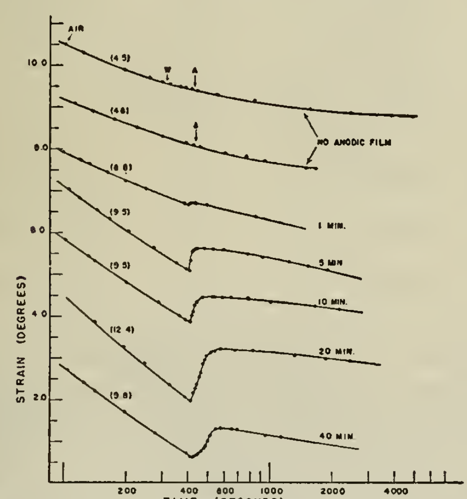


FIGURE 6. Effect of anodizing time at 2.5 volts on after-effect curves of cold worked cadmium.

only a very slight reversal. All others displayed an appreciable reversal of the same order of magnitude, although the longer the anodizing time the longer was the time during which the reversal took place.

The two nonanodized runs were identical, except that as an additional control the first run (No. 115) was twisted and released in air and distilled water first added at 300 seconds. This treatment had no evident effect on the results. The data for these runs are tabulated in Table I.

TABLE I

EFFECT OF FILM THICKNESS

Run no.	Anodizing time	S_1*	R†	t‡
115	0	4.5	—	—
115A	0	4.6	—	—
132	1 min	6.0	0.6	(very short)
128	5 min	9.5	5.4	10
129	10 min	9.5	5.8	20
116D	20 min	12.4	12.5	55
130	40 min	9.8	6.8	95

*S_1, Initial strain rate expressed in tenths of a degree recovered between 200 and 400 seconds.
†R, Magnitude of reversal in tenths of a degree.
‡t, Time, in seconds, for 2/3 of R to occur.

These results show that a thin film increases the initial strain rate considerably, but an additional increase of the film thickness does not further increase the initial slope, but does significantly increase the time for reversal to occur.

Effect of Holding Time on Normal After-Effect

Since the "clean" wire did not behave like the anodized wire, it was found necessary to establish the character of the normal after-effect occurring in a wire without the applied film. A suitable variable for examining the relaxation of a wire, independent of the surface condition, was found to be the time of holding in the twisted condition prior to release.

A group of nonanodized specimens were twisted in distilled water, held for various lengths of time and released. The holding time was computed from commencement of twist to release of load. The plot of strain *versus* logarithm of elapsed time after release of load, Figure 7, indicated that the initial strain rate varied with the time held. In all cases the resultant plot is not exactly a straight line, but rather an S-shaped curve which would extrapolate to zero slope at zero time and at infinite time. The central portion may appear linear for a relatively long period of time. However, to be strictly comparable, the slopes must be computed during the same time interval, in this case between 200 and 400 seconds. Examination of the slopes for various holding times in Figure 7 shows that, in the seg-

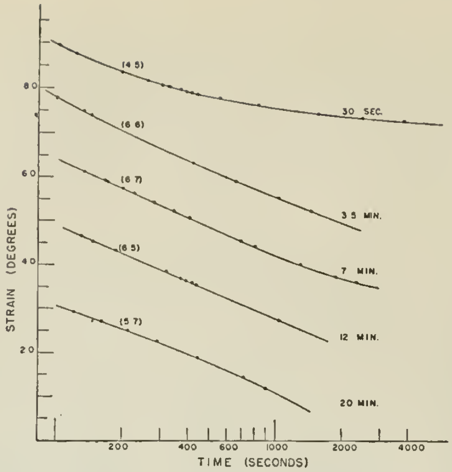


FIGURE 7. Effect of holding time on after-effect curves of non-anodized wires. Holding time was computed from commencement of twist to release of load.

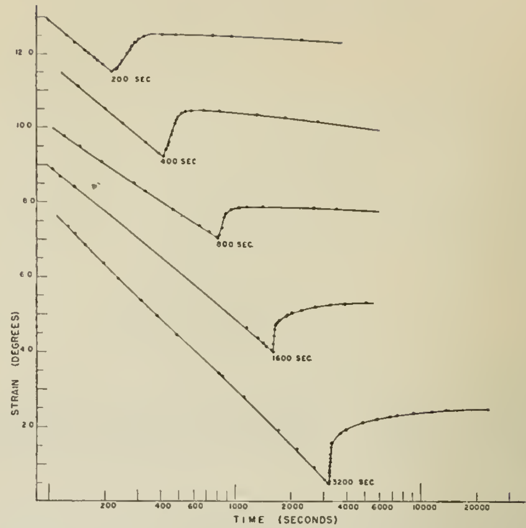


FIGURE 9. Effect of time of replacing water with acid on after-effect curves.

ments plotted, the first three slopes are decreasing in value with increasing holding time, the 12-minute curve is almost linear, and the 20-minute curve increases in value. It appears, then, that these curves have typical anelastic characteristics: increasing the holding time has the effect of moving the inflection point to later time. The slopes during identical time intervals first increase and then decrease with increasing holding time as shown in Figure 8.

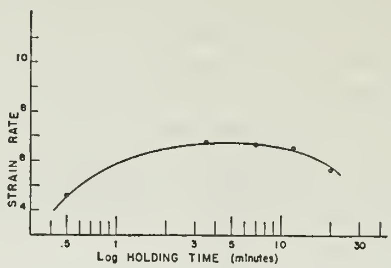


FIGURE 8. Effect of holding time on strain rate of non-anodized wires.

Effect of Delayed Etching Time

The experiments performed by Barrett [3; 4] indicate that the reversal should die out if removal of the film is delayed. Investigation along this line failed to verify this finding. Tests were made using cold-worked cadmium in the standard manner except that the time of replacing water with acid was varied from 200 to 3200 seconds. The resultant data are shown in Figure 9. The amount of reversal and the time during which reversal continued increase with increasing time at which the film is removed. It is especially noteworthy that when the acid was added at 3200 seconds the run was continued for another six hours at the end of which time the reversal still had not died out. The results of this series are tabulated in Table II. Since the

TABLE II

EFFECT OF DELAYED ETCHING TIME

Run no.	Time of adding acid (seconds)	S_1		R	R/S_1	t
127	200	11.8		10.0	.85	57
116A	400	12.6		9.5		
B	400	13.4	$\overline{1.27}$	13.9	$\overline{11.7}$ $\overline{.92}$	$\overline{60}$
C	400	12.4		11.8		
D	400	12.4		12.5		
124	800	10.5		9.5	.91	60
125	1600	12.0		14+	1.17	286
126	3200	14.5		20+	1.38	550

initial slope (S_I) varied slightly throughout these runs it was felt that the ratio of the amount of reversal (R) to the initial slope was more significant than R alone. The amount of reversal, the ratio of reversal to initial slope and the time during which reversal occurred all increase with increasing time of adding acid.

The Inverse Abnormal After-Effect

It has been amply demonstrated that the removal of a film, applied prior to twisting, caused a reversal of the normal after-effect. As a final

critical test of the effect of a film, the order of oxidizing and twisting was inverted by applying the anodic film to the twisted wire while maintaining all other variables constant.

A cylindrical platinum mesh cathode was inserted in the flow tube, and a specimen was mounted in the apparatus in the nonanodized condition. The specimen was then twisted 180 degrees in the usual manner and held for 12 minutes. During this time the flow tube was filled with sodium hydroxide and the specimen anodized at 2.5v for 10 minutes. Upon completion of anodizing the flow tube was drained, flushed, and filled with distilled water. At the end of the 12 minutes the load was released and the recovery noted. At 400 seconds the water was replaced with acid. The effect was opposite to that previously experienced upon etching. Upon attack with acid, the rate of untwisting *increased*, rapidly at first, and then settled down to a rate which was *greater* than the original rate. Two of these runs are plotted in Figure 10 as curves -A- and -B-.

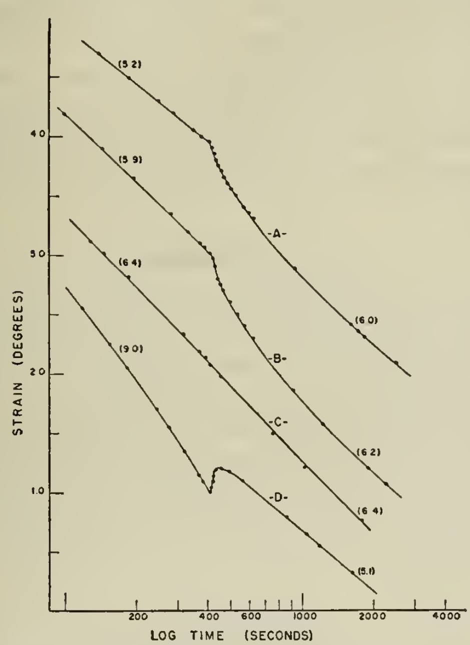


FIGURE 10. Effect of inverting order of twisting and anodizing on after-effect curves. Curves –A– and –B– represent wires which were first twisted, then anodized, then released; curve –C–, twisted and released without anodizing; curve –D–, anodized first, then twisted and released.

As control runs, tests were made on wires treated in the following manner. The specimens were mounted in the apparatus as above. They were then anodized for 10 minutes in the apparatus. Following anodization the sodium hydroxide was replaced with water. The specimens were twisted 180 degrees, held for 12 minutes and released. One of these runs is plotted as curve -D- in Figure 16. Its initial slope was greater than in the runs described above, and when the acid was admitted the "normal 'abnormal after-effect' " occurred, involving a reversal and settling down to a lesser slope. As a final control, a nonanodized wire was held in the twisted position for 12 minutes and then released. Its plot (Curve -C-) shows an intermediate slope corresponding to that finally assumed by -A-, -B- and -D-, after removal of the film. The reproducibility here was quite good and the trend of relaxation immediately after adding the acid very pronounced, as summarized below:

1. Anodizing after twist: Low initial strain rate, increasing upon acid attack.
2. No anodizing: Intermediate strain rate, no effect upon acid attack.
3. Anodizing before twist: High initial strain rate, decreasing upon acid attack.

Discussion

The preceding experiments appear to demonstrate conclusively that the abnormal after-effect is a reproducible phenomenon, directly dependent on the presence and the elastic properties of a film on the surface of a plastically deformed wire. Where the present work duplicates that of Barrett, general agreement is found except with respect to the effect of the time at which the film is removed from the surface. The data obtained show that the latter effect is contrary to that indicated by Barrett.

The phenomena may be accounted for in detail on the basis of a simple mechanical system composed of the plastically deformed wire surrounded by a coherent, elastic film. In the absence of the film, the wire untwists at its characteristic anelastic rate which is determined by the temperature and metallurgical history. If, however, the wire is coated with a coherent, elastic film, the stress resulting from the initial twist is such that the untwisting of the wire is accelerated as shown by the comparison of normal after-effect rates for unoxidized and oxidized wires (Figure 2). A graphic analogy may be drawn between the untwisting wire and the hand of a clock which proceeds at its own regulated speed. If the hand were made of rubber, an externally applied torque would displace the hand to an advanced position; when the external force is removed the hand returns to the position it would have attained without the external force.

In the present case the normal recovery of the wire is forced by the elastic stress imposed by the film to proceed at a rate greater than normal. When the film is removed the wire reverts to a strain and strain rate it would have had in the absence of the film, assuming equal initial conditions. This requires a reversal of the untwisting motion which is the observed abnormal after-effect. In order to cause an elastic reversal of .5 degree, an hydroxide film one micron thick must withstand a stress of 10^9–10^{10} dynes/cm^2. This stress is less than that computed by Orowan [6] (10^{11} dynes cm^2) as the theoretical limit, and is similar to that found by Roscoe [7], Phillips and Thompson [8], and Coffin and Weiman [9] in bending and creep experiments.

The wire does not adopt the new strain instantaneously because a finite time is required to remove the film; in fact, about 60 seconds for the films used in these experiments. Furthermore, the wire may have so far exceeded its normal recovery due to the maintenance of stress in the elastic film that it is plastically deformed and approaches the equilibrium position in a time-dependent manner from the opposite direction. This is demonstrated in the case of delayed removal of the film where reversal continues for a prolonged period after the film is removed, and the period of reversal increases with extended delay in removing the film (Figure 9).

The final test involving an inversion of the order of twisting an anodizing (Figure 10) is a clear demonstration of the effect of the properties of the film and fully conforms with the preceding mechanical description of the observed phenomena. The normal recovery is, in this case, retarded by the initially unstressed film which is stressed elastically during untwisting and so behaves as a brake on the normal rate. Removal of the film permits the wire to accelerate to its normal rate which, in this particular experiment, is that of the unoxidized wire.

Clearly, the observed facts may be accounted for by the simple mechanical concept of a two-component mechanical system comprised of the anelastically recovering metal and its elastically stressed film. The equivalency of this explanation with the dislocation model proposed by Barrett lies in the relation of a surface barrier to plastic deformation.

The torsional plastic deformation generates dislocations which are able to move through the structure of the metal, even in a cold-worked condition, but which accumulate beneath a surface film which is elastically harder than the adjacent metal. These dislocations, if released, would contribute to the permanent or plastic, torsional, deformation of the wire. The normal after-effect is not caused by the migration of these surface-accumulated dislocations, but is a function of the cold-worked condition of the wire and is an internal relaxation occurring within the multitude of interior grains in an anelastic fashion. The dislocations, then, remain adjacent to the surface barrier in a field of high strain energy. This stress concentration caused by the accumulation of dislocations may be thought of as the atomistic analogue of the elasticity of the film. When the film is dissolved, the dislocations are released, and the wire undergoes further plastic deformation in the direction of original twist. The release of dislocations, then, in the mechanical picture, appears as the abnormal after-effect. The fact that the abnormal after-effect does not diminish in magnitude as a result of delay in the removal of the film (Figure 9) is a demonstration that the dislocations remain concentrated at the surface.

Since no attempt has been made here to explain the normal after-effect itself in terms of dislocations, a dislocation analogue to account for the influence of a surface film on the initial strain rate cannot be given.

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